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(Reaffirmed 2003)

पैट्रोलियम तथा इसके उत्पादों के परीक्षण की पद्धति

[पी : 33]

बम पद्धति द्वारा सल्फर

(दूसरा पुनरीक्षण)

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 33]

SULPHUR BY BOMB METHOD

(Second Revision)

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Price Group 2

Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee, PCD 1

FOREWORD

This Indian standard [P : 33] (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 33]

SULPHUR BY BOMB METHOD

(Second Revision)

1 SCOPE

1.1 This method describes the procedure for the determination of sulphur in petroleum products, including lubricating oils containing additives, additive concentrates, and lubricating greases, that cannot be burned completely in a wick lamp. The method is applicable to any petroleum product sufficiently low in volatility that it can be weighed accurately in an open sample boat and containing at least 0·1 percent sulphur.

1.2 This method is not applicable to samples containing elements that give residues, other than barium sulphate, which are insoluble in dilute hydrochloric acid solutions and would interfere in the precipitation step. These interfering elements include iron, aluminium, calcium, silicone and lead which are sometimes present in greases lube oil additives, or additive oils. Other acid insoluble materials that interfere are silica, molybdenum disulphide, asbestos, mica, etc. The method is not applicable to used oils containing wear metals, and lead or silicates from contamination. Samples that are excluded may be analyzed by the method ASTM D 1552 test for sulphur in petroleum products (High-temperature method).

2 REFERENCES

2.1 The following Indian standards are the necessary adjuncts to this standard:

IS No.	*Title*
265 : 1987	Hydrochloric acid (*third revision*)
1083 : 1978	Industrial white oils (*first revision*)
1448 [P : 6] : 1984	Calorific value by bomb calorimeter method (*first revision*)

3 OUTLINE OF THE METHOD

3.1 The sample is oxidized by combustion in a bomb containing oxygen under pressure. The sulphur, as sulphate in the bomb washings, is determined gravimetrically as barium sulphate.

Caution — Strict adherence to all of the provisions prescribed hereafter ensures against explosive rupture of the bomb, or a blow-out, provided the bomb is of proper design and construction and in good mechanical condition. It is desirable, however, that the bomb be enclosed in a shield of steel plate at least 13 mm thick, or equivalent protection be provided against unforseeable contingencies. Initial testing and periodic examination of the bomb is essential, to ensure its fitness for service. This is particularly important if the bomb has been dropped and has any obvious signs of physical damage.

4 SIGNIFICANCE AND USE

4.1 This test provides a means of monitoring the sulphur level of various petroleum products and additives. This knowledge can be used to predict performance, handling or processing properties. In some cases, the presence of sulphur compounds is beneficial to the product and monitoring depletion of the sulphur compounds can be useful; in other cases, the presence of sulphur compounds is detrimental to the processing or use of the product. Some regulatory statutes restrict the concentration of sulphur allowed in some petroleum products.

5 APPARATUS

5.1 Bomb

In good mechanical condition, having a capacity of not less than 300 ml, so constructed that it will not leak during the test and the quantitative recovery of liquids from the bomb will be readily achieved. The inner surface of the bomb may be made of stainless steel or any other material that will not be affected by the combustion process or products. Materials used in the bomb assembly, such as head gasket and electric wire insulation, shall be resistant to heat and chemical action and shall not undergo any reaction that will affect the sulphur content of the liquid in the bomb. A bomb prescribed in IS 1448 [P : 6] is suitable.

5.2 Oxygen Charging Equipment

The valves, gauges, filling tube and fittings used in the oxygen charging system shall meet industry safety codes and shall be rated for use at input pressures up to 206 bar and discharge pressures up to 55 bar. Separate gauges should be provided to show the supply pressure and the bomb pressure. The bomb gauge should be not less than 75 mm in diameter and preferably graduated from 0 to 55 bar (atm) in 1 bar (atm) subdivisions. Both gauges shall be absolutely oil-free and shall never be tested in a hydraulic system containing oil. The charging equipment should include either a pressure reducing valve which will limit the discharge pressure to a maximum of 40 bar or a relief valve set to discharge at 40 bar in case the bomb should accidentally be overcharged. Means should also be provided for releasing the residual pressure in the filling tube after the bomb valve has been closed.

5.3 Sample Cup

Made of platinum, 24 mm in outside diameter at the bottom, 27 mm in outside diameter at the top, 12 mm in height outside and weighing 10 to 11 g.

5.4 Firing Wire

Made of platinum and approximately 0·40 mm in diameter.

5.5 Ignition Circuit

Capable of supplying sufficient current to ignite the cotton wicking or nylon thread without melting the wire. The current should not be drawn from the powerline, and the voltage should not exceed 25 volts.

Caution — The switch in the ignition circuit shall be of a type which remain open, except when held in closed position by the operator.

6 REAGENTS AND MATERIALS

6.1 The following reagents and materials shall be used for the test:

a) *Barium Chloride Solution* (85 g *per litre*) — Dissolve 100 g of barium chloride dihydrate ($BaCl_2.2H_2O$) in distilled water and dilute to 1 litre.

b) *Bromine Water* (*saturated*)

c) *Hydrochloric Acid* (*r.d.* 1·18) — concentrated hydrochloric acid (HCl) (*see* IS 265 : 1987).

d) *Oxygen* — free of combustible material and sulphur compounds, available at a pressure of 40 atm (40 bar).

e) *Sodium Carbonate Solution* (50 g *per litre*) — Dissolve 50 g of sodium carbonate anhydrous (Na_2CO_3) or 135 g of hydrated sodium carbonate ($Na_2CO_3 . 10H_2O$) in distilled water and dilute to 1 litre.

f) *White Oil* — Liquid Paraffin (*see* IS 1083 : 1978).

g) *Cotton Wicking Nylon Sewing Thread* — white.

7 PROCEDURE

7.1 Preparation of Bomb and Sample

7.1.1 Cut a piece of firing wire 100 mm in length. Coil the middle section (about 20 mm) and attach the free ends to the terminals. Arrange the coil so that it will be above and to one side of the sample cup. Insert between two loops of the coil a wisp of cotton or nylon thread of such length that one end will extend into the sample cup. Place about 5 ml of Na_2CO_3 solution in the bomb (*see* Note 1) and rotate the bomb in such a manner that the interior surface is moistened by the solution. Introduce into the sample cup the quantities of samples and white oil (*see* Note 2 and 3) specified in Table 1, weighing the sample to the nearest 0·2 mg (when white oil is used, stir the mixture with a short length of quartz rod and allow the rod to remain in the sample cup during the combustion).

Table 1 Mass of Sample and White Oil

(*Clause* 7.1.1),

Sulphur Content, Percent (1)	Mass of Sample, g (2)	Mass of White Oil (3)
5 or under	0·6 to 0·8	0·0
Over 5	0·3 to 0·4	0·3 to 0·4

NOTES

1 After repeated use of the bomb for sulphur determinations, a film may be noticed on the inner surface. This dullness should be removed by periodic polishing of the bomb. A satisfactory method for doing this is to rotate the bomb in a lathe at about 300 rpm and polish the inside surface with emery polishing paper Grit No. 2/0, or equivalent paper, coated with a light machine oil to prevent cutting, and then with a paste of grit-free chromic oxide and water. This procedure will remove all but very deep pits and put a high polish on the surface. Before using the bomb, it should be washed with soap and water to remove oil or paste left from the polishing operation.

Caution — Do not use more than 1·0 g total of sample and white oil or other low sulphur combustible material, or more than 0·8 g.

2 Use of sample mass containing over 20 mg of chlorine may cause corrosion of the bomb. To avoid this, it is recommended that for samples containing over 2 percent chlorine, the sample mass be based on the chlorine content as given in the following table:

Chlorine Content, Percent	*Mass of Sample, g*	*Mass of White Oil, g*
2 to 5	0·4	0.4
Over 5 to 10	0·2	0·6
Over 10 to 20	0·1	0·7
Over 20 to 56	0·05	0·7

3 If the sample is not readily miscible with white oil, some other low sulphur combustible diluent may be substituted. However, the combined mass of sample and non-volatile diluent shall not exceed 1·0 or 0·8 g by mass.

7.2 Addition of Oxygen

7.2.1 Place the sample cup in position and arrange the cotton wisp or nylon thread so that the end dips into the sample. Assemble the bomb and tighten the cover securely.

Caution — Do not pass oxygen or ignite the sample if the bomb has been jarred, dropped or tilted. Admit oxygen slowly (to avoid blowing the oil from the cup) until a pressure is reached as indicated in Table 2.

7.3 Combustion

7.3.1 Immerse the bomb in a cold distilled-water bath. Connect the terminals to the open electrical circuit. Close the circuit to ignite the sample.

Caution — Do not go near the bomb till at least 20 seconds after firing. Remove the bomb from the bath after immersion for at least 10 minutes. Release the pressure at a slow, uniform rate such that the operation requires not less than 1 minute. Open the bomb and examine the contents. If traces of unburned oil or sooty deposits are found, discard the determination and throughly clean the bomb before again putting it in use (*see* Note under **7.1.1**).

Table 2 Gauge Pressure

(*Clause* 7.2)

Capacity of Bomb, ml	Minimum Gauge Pressure* atm or bar	Maximum Gauge Pressure* atm or bar
(1)	(2)	(3)
300 to 350	38	40
351 to 400	35	37
401 to 450	30	32
451 to 500	27	29

*The minimum pressures are specified to provide sufficient oxygen for complete combustion and the maximum pressure represent a safety requirement.

7.4 Collection of Sulphur Solution

7.4.1 Rinse the interior of the bomb, the oil cup, and the inner surface of the bomb cover with a fine jet of distilled water, and collect the washings in a 600 ml beaker having a mark to indicate 75 ml. Remove any precipitate in the bomb by means of a rubber policeman. Wash the base of the terminals until the washings are neutral to a suitable indicator. Add 10 ml of bromine water to the washings in the beaker. (The volume of the washing is normally in excess of 300 ml.) Place the sample cup in a 50 ml beaker. Add 5 ml of saturated bromine water, 2 ml of hydrocholoric acid (HCl), and enough distilled water just to cover the cup. Heat the contents of the beaker to just below its boiling point for 3 or 4 minute and add to the beaker containing the bomb washings. Wash the sample cup and the 50 ml beaker thorougly with distilled water. Remove any precipitate in the cup by means of a rubber policemen. Add the washings from the cup and the 50 ml beaker, and the precipitate, if any, to the bomb washings in the 600 ml beaker. Do not filter any of the washings, since filtering would remove any sulphur present as insoluble material.

7.5 Determination of Sulphur

7.5.1 Evaporate the combined washings to 200 ml on a hot plate or other source of heat. Adjust the heat to maintain slow boiling of the solution and add 10 ml of barium chloride ($BaCl_2$) solution, either in a fine stream or dropwise. Stir the solution during the addition and for 2 minutes thereafter. Cover the beaker with a fluted watch glass and continue boiling slowly until the solution has evaporated to a volume of approximately 75 ml as indicated by a mark on the beaker. Remove the beaker from the hot plate (or other source of heat) and allow it to cool for 1 h before filtering. Filter the supernatent liquid through an ashless, quantitative filter paper (*see* Note 1). Wash the precipitate with water, first by decantation and then on the filter until free from chloride. Transfer the paper and precipitate to a weighed crucible and dry (*see* Note 2) at low heat until the moisture has evaporated. Char the paper completely without igniting it, and finally ignite at a bright red heat until the residue is white in colour. After ignition is complete allow the crucible to cool to room temperature and weigh.

NOTES

1 A weighed porcelain filter crucible (Selas type) of 5 to 9 μ m porosity may be used in place of the filter paper. In this case the precipitate is washed free of chloride and then dried to constant mass at 500 + 25 °C.

2 A satisfactory means of drying, charring and igniting the paper and precipitate is to place the crucible containing the wet filter paper in a cold electric muffle furnance and to turn on the current. Drying, charring and ignition usually will occur at the desired rate.

7.6 Blank Determination

7.6.1 Make a blank determination whenever new reagents, white oil or other low-sulphur combustible material are used. When running a blank on white oil, use 0·3-0·4 g and follow the normal procedure.

8 CALCULATION

8.1 Calculate the sulphur content of the sample as follows:

$$\text{Sulphur, percent by mass} = \frac{(A-B)\ 13{\cdot}73}{M}$$

where

A = mass of barium sulphate ($BaSO_4$) in g obtained from sample,

B = mass of barium sulphate ($BaSO_4$) in g obtained from blank, and

M = mass of sample in g used.

9 REPORTING

9.1 Report the result of the test to the nearest 0·01 percent as total sulphur.

10 PRECISION

10.1 Results of duplicate tests shall not differ by more than the following amounts (*see* Note):

Sulphur, percent by mass	*Repeatability*	*Reproducibility*
0·1 to 0·5	0·04	0·05
Over 0·5 to 1·0	0·05	0·09
Over 1·0 to 1·5	0·08	0·15
Over 1·5 to 2·0	0·12	0·25
Over 2·0 to 5·0	0·18	0·27

NOTE — The precision shown in the above table does not apply to samples containing over 2 percent chlorine because an added restriction on the amount of sample which may be ignited is imposed.

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